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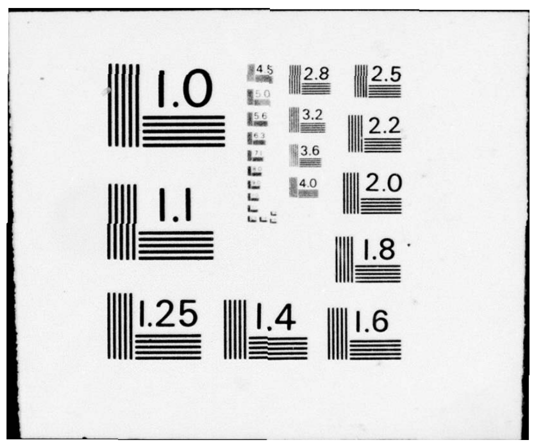
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PRODUCTION ENGINEERING MEASURES TO MANUFACTURE SUPER FINE FINISH BERYLLIA

FINAL REPORT
30 June, 1974 to 22 September, 1976

CONTRACT NO. DAAB07-74-C-0606

U.S. ARMY ELECTRONICS COMMAND

Fort Monmouth
New Jersey

RESEARCH AND DEVELOPMENT

BRUSHWELLMAN

ENGINEERED MATERIALS

ELMORE, OHIO 43416

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A progressive polishing process was developed to reduce the roughness of the working surface of beryllia substrates to less than four microinches center line average. Commercially available 1 inch x 1 inch and 2 inch x 2 inch beryllia plates containing 99.5 percent beryllia served as the input material. To minimize warpage, one face of the beryllia plate was machined on a polishing wheel charged with six micron diamond paste. The opposite or working surface was polished on the same wheel to a surface finish of 5-8 microinches. The cleaned working surface was final polished on a polishing over

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20. Abstract

wheel charged with three micron diamond paste. Scale-up from a twelve inch diameter laboratory polisher to a twenty-four inch diameter manufacturing unit was demonstrated. Dicing was used to produce small 0.080 inch x 0.110 inch substrates from polished 1.0 inch x 1.0 inch plates. A Pilot Run demonstration achieved the production rate goal of 1000 substrates per week for each of three sizes of beryllia substrates.

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BRUSH WELLMAN INC.
ELMORE, OHIO 43416

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PRODUCTION ENGINEERING MEASURES
TO MANUFACTURE
SUPER FINE FINISH BERYLLIA

FINAL REPORT
30 June, 1974 to
22 September, 1976

The object of this program is to develop the production engineering measures to manufacture beryllia substrates for which the working surface of the substrates shall not exceed a four microinch center line average surface finish.

CONTRACT NO.: DAAB07-74-C-0606

Prepared by: Kenneth A. Walsh

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SECTION I

ABSTRACT

A two-step progressive polishing process was developed to reduce the surface roughness of beryllia substrates to less than four microinches center line average. Commercially available beryllia plates containing 99.5 percent beryllia could be used as the input material. Scale-up from a twelve inch diameter laboratory polisher to a manufacturing-scale twenty-four inch diameter polisher was demonstrated. Dicing was used to produce the small 0.080 inch x 0.110 inch substrates from a polished 1.0 inch x 1.0 inch plate. A Pilot Run demonstration achieved the production rate objective of 1000 substrates per week of each of three sizes of beryllia substrates. Literature values, which served as the basis for Specification SCS-472, were generally confirmed. An exception was the dielectric constant which required a minor revision of the minimum-maximum range to 6.6 - 6.8, especially for the higher test frequencies of 1 GHz and 10 GHz. The volume resistivity values generated in this program also required revision from 10^{14} to 10^{13} ohm-cm minimum specification when measured at 100°C.

SECTION II

PURPOSE

The purpose of this program is to develop the production engineering measures to manufacture beryllia substrates of which the working surface shall not exeed a four microinch surface smoothness by progressive polishing techniques. The input material will be commercially available beryllia, the composition of which shall not be less than 99.5% BeO.

SECTION III

INTRODUCTION

The work performed under Contract No.DAAB07-74-C-0606 included the preparation of Engineering Samples, delivery of First Article units produced on manufacturing-scale equipment, and a Pilot Run demonstration of the capability of producing 1000 units/week of each of three substrate sizes by progressive polishing. The input material was commercially available Thermalox 995 beryllia plates, the composition of which is 99.5% BeO or greater. The objective of the program was met in polishing the working surface of the beryllia substrates to a four microinch surface smoothness or less.

The scale-up from Engineering Samples prepared on a laboratory polisher to the First Article and Pilot Run units produced on manufacturing-scale equipment was accomplished with a minimum of problems with no major changes in the process.

Test data were obtained on the manufactured samples or on special test specimens made from the same lot of beryllia powder and subjected to the same thermal history. Testing included the measurement of physical, electrical, and thermal properties of the polished substrates or the special test specimens.

SECTION IV

PROCESS DEVELOPMENT

4.1 Device

Since the composition of the substrate product shall not be less than 99.5% beryllia, commercially available Thermalox 995 beryllia or its equivalent is required for the input material. Chemically, Thermalox 995 contains a minimum of 99.5% beryllium oxide with the principal impurities being additions of magnesium and silicon oxides. Commercially available 1.0 in. x 1.0 in. and 2.0 in. x 2.0 in. beryllia plates have a surface roughness of 10-15 microinches in the as-fired condition. Warpage up to 0.004 inches per linear inch exists as a deterrent to polishing the entire surface.

Cylindrical surfaces of gas bearing parts had been machined using diamond wheel grinding and lapping methods for M.I.T. Charles Stark Draper Laboratory. Four machine houses had obtained cylindrical surfaces with 4.0 microinch center line average (CLA). Flat surfaces of gas bearing parts had been similarly machined to a finish of CLA $\leq$ 2.0 microinches by one machine shop. This gave evidence of the technical feasibility of polishing substrates, but no indication of the processing costs was available.

The polished substrates are classified as Type A, Type B, or Type C depending on the dimensions shown in Table I.

TABLE I

DIMENSIONS OF SUBSTRATES

All dimensions in inches

Type	Length	Width	Thickness
A	0.080±.003	0.110±.003	0.010±.002
B	1.0±.005	1.0±.005	0.025±.002
C	2.0±.010	2.0±.010	0.040±.003

The as-fired warpage in beryllia plates is removed by lapping or machining the surfaces flat and parallel. This is a destructive and regressive step which degrades the surface finish to 20-35 microinches, depending on the coarseness or grit of the abrasive used. This state-of-the-art limitation was eliminated by first polishing the non-working surface with six micron diamond paste. The plates were then inverted in the polishing ring and the working surface was polished with six micron diamond paste. Both surfaces had a surface finish of 5-6 microinches. Polishing of the working surface with three micron diamond paste achieved the objective of the program. Consequently, the amount of material to be removed and polishing time were decreased, e.g., thickness of the input plate for Type C substrates was decreased from 0.047 in. to 0.044 in. in the course of the process development. Dimensions of the as-fired input beryllia plates are given in Table II. Type A parts were produced by dicing or sawing of 1.0 in. x 1.0 in. polished plates, each of which yields 63 Type A substrates. Type B input plates or substrates rejected for sub-dimensional thickness could be lapped or machined to supply Type A input plates to improve the overall yield.

TABLE II

DIMENSIONS OF INPUT PLATES

All dimensions in inches

Type	Length	Width	Thickness
A	1.0±.005	1.0±.005	0.013±.002
B	1.0±.005	1.0±.005	0.029±.002
C	2.0±.010	2.0±.010	0.044±.003

As-fired Thermalox 995 is polycrystalline beryllia at 95-96% of theoretical density. It is available with an average grain size of 8, 12, or 18 microns. Ten samples of each grain size were polished with six micron diamond paste. The average surface finish was five microinches for each of the three grain sizes, eliminating this variable from further consideration. The 12 micron grain size Thermalox 995 plate was selected, therefore, because it is the standard and slightly less costly input material for polishing. Grain size is not affected by polishing, so the product substrates have grain sizes of 10-12 microns.

4.2 Equipment and Tooling

Lapping and polishing tests to develop the process were performed on a twelve inch diameter unit manufactured by Speedfam Inc. The Speedfam 12 is 20 in. x 16 in. and 14 in. high . It has an electric motor and drive mechanism rotating a 12 in. diameter wheel and up to four 4 1/2 in. inside diameter rings containing the parts to be polished. The rings are driven from a wheel on the center shaft; they remain stationary but rotating by four idler wheels. The machine is equipped with an electric timer and switch. The wheel speed is 87 rpm, which was fixed by the manufacturer as the compromise between material removal rate and loss of abrasive paste by centrifugal action. The rotating wheels or laps are made of steel with a spiral groove surface design for use with diamond paste. Because the diamond tends to impregnate the relatively soft spiral groove wheel, the wheel must be used for one grit of abrasive only. Since the Type A input plates are thinner and more fragile than Type B and Type C parts, special handling is required. The Speedfam 12 unit is used for production of Type A substrates to minimize breakage losses.

A Speedfam unit with a 24 in. diameter polishing wheel is installed at Brush Wellman Inc. for lapping of beryllia parts to improve surface flatness or metalizability. The Speedfam 24 has four rings for work holders, each ring in a quadrant of the polishing wheel. Each ring holds 52 of the Type B plates giving a capacity for 208 substrates per polishing cycle. Fully loaded with Type C plates gives 48 substrates per cycle. Each ring rotates about its axis while the spiral groove polishing wheel rotates independently at 87 rpm. The applied pressure can be varied with four adjustable hydraulic rams.

For dicing of the polished 1.0 in. x 1.0 in. x 0.010 in. substrate, the parts are mounted on a master saw board by heating and melting DeKhotinsky cement, positioning and aligning the parts, and cooling the cement. A Brown and Sharpe Surface Grinder or a Do-All Grinder is equipped with a seven inch diameter diamond edge saw blade with a feed rate of 2.5 inches per minute at a machine speed of 3200 rpm.

The Pilot Run was performed in an existing facility of Brush Wellman Inc. The polishing area is 17 ft. x 23 ft. in which the Speedfam 12 and Speedfam 24 units are located. The area is also occupied by a Speedfam 32 polishing unit, but the floor space would not be decreased much by excluding it. Four working tables for cleaning the substrates and for performing in-process inspection are located in this working area.

The existing dicing area is adjacent to the lapping and polishing area of the Brush Wellman Inc. facility. It has a group of hot plates on a table for mounting the polished substrates on the master saw board. The same equipment was used for removal of diced substrates. An area for cleaning parts after dicing provides a sink and table with an air dryer.

The Pilot Run dicing was performed on a Do-All Grinder in a hooded area equipped with the necessary ventilation. The hood also contains a Brown and Sharpe Surface Grinder, which is also capable of performing the dicing operation. A work table for in-process measurements and inspections is available. Floor space in the dicing area is 14 ft. x 23 ft., including aisles for movement of material and personnel.

Replacement costs for the polishing and dicing equipment used in the Pilot Run include:

Speedfam 12 with 2 spiral wheels	$1,985
Speedfam 24 with 2 spiral wheels	13,835
Do-All Grinder	24,500
Master saw board table & hot plates	500
Cleaning tank or sink and tables	300
Hood to central ventilation	2,500
TOTAL	$43,620

The total cost of $43,620 does not include any portion of the central ventilation, scrubbers, and bag collectors for protection of employees, nor the cost of that portion of the building in which the polishing and dicing areas are installed. All equipment and facilities used in the Pilot Run are owned by Brush Wellman Inc.

4.3 Process Variables

The original concept of the processing steps to manufacture super fine finish beryllia included the following:

A. Lap or machine input plates to provide flat, parallel surfaces.

B. Polish to restore as-fired surface finish.

C. Polish with 6 micron diamond paste.

D. Polish with 3 micron diamond paste.

E. Polish with 1 micron diamond paste.

F. Dicing of Type A substrates.

Lapping pressure and time, along with the grit size and material in the abrasive, were variables in removing material to obtain flat, parallel surfaces. With 240 and 320 grit silicon carbide and with 320 grit boron carbide abrasives the rate of removal of beryllium oxide was about 0.0015$\pm$.0005 in./min. independent of the pressure from weights added to the polishing ring. Lapping with these abrasives increased the cost of beryllium oxide lost to waste and required tight control of the lapping time to obtain the required thickness of the substrates. The coarse abrasives had the adverse effect of increasing the surface roughness to 30-40 microinches.

Attempts to combine Step A and Step B were made with 600 grit silicon carbide and with 15 micron diamond polishing abrasives. For 600 grit silicon carbide abrasive, the rate of removal of beryllium oxide was 0.00025 in./min. Lapping pressures of 4-6 psi were used to obtain coverage of the entire surface of the beryllia plate. Surface roughness was degraded to 18-23 microinches. Increases in polishing times/Step to 10-15 min. were not considered excessive, since Steps A and B could be combined. With 15 micron diamond polishing abrasive, similar polishing pressures and time gave removal rates of 0.0004 in./min. Surface roughness was 18-22 microinches.

Subsequently, flat plates with 18-23 microinch surface roughness were polished with six micron diamond polishing abrasive. Polishing pressure was not a significant variable influencing the surface finish. The effect of polishing time on surface finish was confounded by the total number of polishing runs with the new polishing wheel. As the total number of runs increased, the rate of removal of beryllia during polishing nearly doubled that in the initial runs. As the polishing wheel became impregnated with diamond particles, the rate of removal of material

stabilized at about 0.0003 in./min. The attained surface finish also improved with age of the polishing wheel. Step C polishing improved the surface finish to 6-8 microinches.

Substrates from Step C polishing at 8 microinch surface finish were polished on a new wheel with three micron diamond paste. Again, the final surface finish improved with age of the wheel. Increasing the polishing pressure to six psi improved the surface finish. Polishing for 10 and 15 minutes gave surface finishes of 3 microinches. Since this surface finish was below the 4 microinch objective of the program, Step E was eliminated from the process.

The 0.0003 in./min. rate of removal of material during polishing with six micron diamond abrasive was sufficient to consider the elimination of Step A and Step B. The input beryllia plates at that time for Type B and Type C substrates required the removal of 0.007 in. from the thickness dimension. Polishing for 15 min. removed 0.0049 in. from the thickness dimension, producing a 6 microinch surface finish. Continuous polishing for 25 min. removed the requisite 0.0074 in. from the thickness dimension and gave a 6 microinch surface finish. Steps A and B were eliminated. Subsequent purchases of input plates for Type B and Type C substrates were made in accordance with the dimensional specifications of Table II, so the polishing time could be reduced to that necessary to remove 0.004 in. from the thickness dimension.

The substrates from Step C polishing only could be polished with 3 micron diamond polishing abrasive to meet the objective of a working surface with a finish of 4 microinches or less. A modification of Step C was adopted to improve process control, improve yields, and to give a slightly smoother surface finish. The plates were polished on the 6 micron diamond

wheel for 10 minutes with a polishing pressure of 1 psi. In this interval, most of the plates were polished to a 6 microinch surface finish, but some of the more highly warped plates did not receive complete surface coverage. The pressure heads were raised and the parts were inverted or flipped to polish the opposite side of the plate. Spot checks of the thickness were used to estimate the rate of material removal. The plates were polished on the opposite side for 10 minutes. Thickness dimensions of a few of the substrates were measured. During these measurements, surface damage by the micrometer is to be avoided. All parts were rearranged within the ring and polishing was continued as long as necessary to obtain a fully polished surface and a thickness below the maximum permissible. The working surface of the substrate had a surface finish of 5-6 microinches and the non-working surface was more parallel to the working surface.

The modified Step C process reduced the polishing time in Step D to 10 minutes. The surface finish was 3-3.5 microinches on all substrates tested from the Pilot Run.

Cleanliness is an important item in a progressive polishing process. Before installing a wheel, the machine table, rings, and heads should be thoroughly cleaned with soapy water. At the conclusion of every polishing run, the substrates should be cleaned immediately in soapy water, or the parts become permanently stained with diamond powder. To aid the cleaning process, water-base diamond paste only should be used. A polishing wheel being removed should be thoroughly cleaned and dried. Storage in sealed plastic bags is recommended.

Lubrication of the polishing wheel before and during a run uses a mixture of 5 parts water and 1 part ethylene glycol by volume. Application of lubricant by spraying should be especially generous for a used wheel just removed from storage and before and during the initial stages of a new series of runs, e.g., the first run of the day with less than three shift operation.

Input plates for the Type A parts were purchased as machined to the 0.013 in. thickness dimension with a 30-35 microinch surface finish. Parts are attached to the face of the weights within the rings with double adhesive tape. The working surface only is polished in Step C with 6 micron diamond abrasive on the Speedfam 12 polishing wheel. After 10 minutes polishing, the parts are checked for the rate of material removal. Polishing is continued as needed to reach the 0.010 in. thickness requirement. After cleaning the parts and the machine, a 3 micron diamond polishing wheel was installed. The substrates were mounted to the weights as before and polished for 10 minutes to provide the surface finish required on the working surface.

Dicing or sawing of substrates with the length and width dimensions of Type A substrates has been frequently performed on thicker plates. With the Brown and Sharpe Surface Grinder or Do-All Grinder equipped with a seven inch diameter diamond edge saw blade, the only question was that of material pull-out as very small chips at the edges of the substrate. To minimize this risk, the polished substrates were mounted on the master saw board with the polished surface upward. The exit direction of the saw is thereby away from the working surface of the substrate. No problems of degradation of the polished surface were detected; the non-working surface was also unharmed.

4.4 Flow Charts of Manufacturing Process

4.4.1 Flow Chart for Type A Substrates

Input 1.0" x 1.0" x .013"	Capacity Per Cycle	Labor Man-Hrs.	Yield %
↓			
Speedfam 12 6 Micron Diamond	40 Plates	2.0	
↓			
Cleaning and Inspection		2.0	95
↓			
Speedfam 12 3 Micron Diamond	40 Plates	2.0	
↓			
Cleaning and Inspection		2.0	95
↓			
Mounting Saw Board	36 Plates	2.0	100
↓			
Do-All Grinder Dicing	2268 Sub- strates 2.5 in./min.	7.0	
↓			
Cleaning and Inspection		4.0	95
↓			
Type A Substrates	2150	21.0	85

Because the Type A material is unusually thin, plate breakage losses occur during attachment of the plates to the weights with double adhesive tape and during their removal for inspection. The attachment-removal from the adhesive occurs twice in the process. Generally, breakage occurs at the corners of the plate, which limits the loss. In the Pilot Run, corner breakage during polishing and dicing caused the loss of 12 Type A substrates from a potential of 252 parts, for a loss of 5%. Yield improvement and input material costs would be favored by increasing the thickness of Type A parts to 0.025 in., providing this would be consistent with their application. Since completing the Pilot Run, a bonding material, Crystalbond 555, soluble in hot water, has been successfully used to replace the tape method of attaching the input plates to the weights of the Speedfam 12 polisher. This substitution was shown to decrease the time for mounting and cleaning of the substrates. This time saving is not incorporated in the Flow Chart for Type A substrates. The use of Crystalbond 555 in the dicing operation has not been demonstrated. It will be tested during the manufacture of similar diced commercial products outside the scope of this program.

With the contract requirement of 250 Type A substrates, the equipment was operated at about 20-25% of capacity in the Pilot Run. This low utilization of capacity still demonstrated a production rate of 1200 substrates per week of five 8-hour days. The Flow Chart shown utilizes one operator for polishing, dicing, and cleaning for shipment of 4000 Type A substrates per week of five 8-hour days. The Pilot Run was performed by one Research and Development technician for polishing and one Manufacturing machinist for the dicing operation.

4.4.2 Flow Chart for Type B Substrates

Input 1.0" x 1.0" x .029"	Capacity per Cycle	Labor Man/Hrs.	Yield %
↓			
Speedfam 24 6 Micron Diamond	208 Plates	0.5	
↓			
Inspection and Reversal		0.75	
↓			
Speedfam 24 6 Micron Diamond	208 Plates	1.50	
↓			
Cleaning and Inspection		1.0	95
↓			
Speedfam 24 3 Micron Diamond	208 Plates	1.0	
↓			
Cleaning and Inspection		1.0	95
↓			
Type B Substrates	187 Sub- strates	5.75	90

With the first load of the Pilot Run, the polishing wheel had been removed from storage after prior use with 6 micron diamond. Insufficient lubricant was applied to the wheel before attaching the rings and insufficient lubricant was used in the initial minutes of polishing. More material was removed than expected in the first 10 minutes. Pressure was reduced to compensate by reducing material removal during 6 micron diamond polishing of the working surface. This change in procedure increased the polishing time to achieve the final thickness dimension. After polishing with 3 micron diamond, twenty-nine parts were below the 0.023 in. thickness minimum. One part exceeded the 0.027 in. maximum and one part had cracked. The 177 acceptable parts represented an 85% yield, in spite of which a production rate of 1770 substrates per week of five work days was demonstrated in the Pilot Run. Proper lubrication should increase the yield to 90% or better and reduce the cycle time, as was demonstrated in a second cycle with 104 plates in two rings. The Pilot Run was performed by one Research and Development technician and one Manufacturing machinist, both of whom had gained experience in the First Article run.

4.4.3 Flow Chart for Type C Substrates

Input 2.0" x 2.0" x .044"	Capacity per Cycle	Labor Man/Hrs.	Yield %
Speedfam 24 6 Micron Diamond	48 Plates	0.33	
Inspection and Reversal		0.17	
Speedfam 24 6 Micron Diamond	48 Plates	0.33	
Cleaning and Inspection		0.50	95
Speedfam 24 3 Micron Diamond	48 Plates	0.50	
Cleaning and Inspection		0.50	95
Type C Substrates	43 Substrates	2.33	90

In the Pilot Run, polishing of Type C plates immediately followed the processing of Type B substrates with both polishing abrasives. Consequently, the polishing wheel was closer to the steady-state condition associated with continuous production. With fewer plates per cycle, cleaning and inspection times during processing were reduced. The 6 micron diamond polishing cycle required 50 minutes plus a 3 micron diamond cycle of 35 minutes. Five cycles per shift were completed. With a demonstrated 90% yield of acceptable parts, the production rate for two operators in a five-day week was 1075 substrates per week. Dimensional losses were nil. Corner breakage through collision of parts in the ring during polishing was responsible for the 10% loss. Reduction of this loss is unlikely, but increases in losses through collision breakage can be limited by frequent inspection and replacement of the felt backing of the plates in the ring. The Pilot Run was performed by one Research and Development technician and one Manufacturing machinist.

4.4.4 Pilot Run

The Pilot Run was completed on the equipment and in the operating area of Ceramics Manufacturing of Brush Wellman Inc. The pilot line is part of the existing and permanent facility described in Section 4.2. Operating personnel consisted of one Research and Development technician and one Manufacturing machinist for the polishing operations and a second Manufacturing machinist for the dicing operation. The Pilot Run to produce 250 each of Type A, Type B, and Type C substrates was completed in five operating days of one shift. Elapsed time was increased by the necessary cleaning

of equipment and changing of wheels between 6 micron and 3 micron diamond polishing and return of the cleaned polishing wheels to storage. Elapsed time was also increased by the inclusion of 100% inspection for surface finish and the thickness dimension for the first complete polishing cycle for each substrate Type, so the government Project Engineer could factor the yield into the production rate. The surface finish of about 260 substrates was measured during the Pilot Run, and 100% had achieved the $<$ 4 microinches center line average objective of the program. Production rates verified by the government Project Engineer were

Type A	1200 substrates/week
Type B	1770 substrates/week
Type C	1075 substrates/week

The contractual requirement of 1000 substrates per week for simultaneous production of the three substrate Types would require one shift operation of the Speedfam 12 unit and the dicing operation, and two shift operation of the Speedfam 24 unit in a five-day week. Productivity per man-hour would be increased by scheduling longer polishing runs between the 6 micron and 3 micron diamond steps.

4.5 Data and Analysis

Representative samples of the polished beryllia substrates or special test specimens, where required, were subjected to testing in accordance with Electronics Command Technical Requirements SCS-472 dated 21 February 1974, "Super Fine Finish Beryllia" as modified, under the terms of the contract.

4.5.1 Dimensions

4.5.1.1 Specification - The dimensions of the substrates shall be as specified in Table I, paragraph 4.1.

4.5.1.2 Length and Width Measurements - Measurements of Type A and Type B substrates are made with a Brown and Sharpe micrometer having a 0-1 in. range, 0.001 in. graduations with a vernier reading to 0.001 inch. Type C substrates are measured with a 2-3 in. range Lufkin micrometer, which has 0.001 in. graduations. For the small Type A substrates only one measurement of length and width is meaningful. The length and width of the Type B and Type C substrates are measured at three points with the low and high readings being recorded.

4.5.1.3 Thickness Measurement - Thickness is measured with the 0-1 in. range Brown and Sharpe micrometer. Type A substrates are measured at one point only, although the 1 in. x 1 in. x .010 in. substrate from which Type A substrates were sliced is monitored with six random readings. Six random readings of thickness of Type B and Type C substrates are made. The lowest and highest values are recorded. During these measurements, care

is exercised to avoid degradation of the polished working surface of the substrate with scratch marks.

4.5.1.4 Type A Substrates Data -48 samples were measured during the program with the results listed in Table III. All substrates met the specification for length, width, and thickness. The variation in length and width follows normal distribution, probably reflecting vibration of the saw blade during dicing. The scatter in thickness can be attributed to the small number of polished plates which were diced and to the warpage within the plates.

4.5.1.5 Type B Substrates Data - 48 samples were measured during the program. Since the substrates are square, length and width results are combined to show the distribution of low and high values in Table IV. All samples met the specification requirements for length, width, and thickness. During the Pilot Run determination of yield, 100% inspection of the 208 parts was made for thickness. One part was oversize and 29 parts were below the 0.023 in. minimum. The oversize and undersize parts were rejected through this in-process inspection. Excessive removal of stock is believed to have occurred because of insufficient lubrication during the first run with a dry wheel impregnated with diamond from previous usage. Undersize parts were not produced in subsequent cycles.

4.5.1.6 Type C Substrates Data - 48 samples were measured during the program. Length and width results are combined to show the low and high values observed. The data are listed in Table V. All specimens met the specification requirements for length, width, and thickness.

TABLE III

DIMENSIONS OF TYPE A SUBSTRATES

Width, in.	Frequency	Length, in.	Frequency	Thickness, in.	Frequency
.0770	1	.1086	1	.0090	1
.0790	1	.1094	1	.0091	3
.0800	2	.1099	1	.0093	3
.0801	1	.1100	8	.0094	2
.0802	2	.1101	8	.0095	2
.0803	3	.1102	10	.0096	1
.0804	8	.1103	8	.0101	1
.0805	10	.1104	1	.0102	1
.0806	12	.1105	2	.0103	3
.0807	4	.1106	1	.0104	2
.0808	1	.1110	1	.0105	5
.0809	1	.1111	1	.0106	4
.0810	2	.1113	2	.0107	1
		.1116	1	.0108	3
		.1117	1	.0109	2
		.1118	1	.0110	2
				.0111	1
				.0112	5
				.0113	1
				.0114	1

TABLE IV

DIMENSIONS OF TYPE B SUBSTRATES

Length, in.	Frequency Low	Frequency High	Thickness, in.	Frequency Low	Frequency High
.996	4	0	.0230	2	0
.997	24	0	.0235	2	0
.998	43	0	.0236	1	0
.999	22	5	.0238	4	0
1.000	3	14	.0240	8	5
1.001	0	43	.0243	3	0
1.002	0	23	.0244	3	0
1.003	0	8	.0245	2	0
1.004	0	3	.0246	2	1
			.0247	0	1
			.0248	3	2
			.0250	6	6
			.0252	3	2
			.0253	2	0
			.0255	3	3
			.0257	2	3
			.0258	1	1
			.0260	1	7
			.0261	0	2
			.0262	0	3
			.0263	0	4
			.0265	0	5
			.0266	0	1
			.0267	0	2

TABLE V

DIMENSIONS OF TYPE C SUBSTRATES

Length, in.	Frequency Low	High
1.991	2	0
1.992	1	0
1.993	1	0
1.994	7	0
1.995	6	1
1.996	14	0
1.997	20	0
1.998	22	4
1.999	13	1
2.000	7	4
2.001	3	9
2.002	0	13
2.003	0	11
2.004	0	12
2.005	0	16
2.006	0	9
2.007	0	8
2.008	0	8

Thickness, in.	Frequency Low	High
.038	4	0
.039	13	4
.040	13	12
.041	13	13
.042	5	11
.043	0	8

4.5.2 Substrate Composition

4.5.2.1 Specification - The composition of the substrate shall not be less than 99.5% beryllia.

4.5.2.2 Test Method - Non-beryllium metallic elements are determined by the d.c. arc carrier distillation method with a Jarrell-Ash Model JA-7101 spectrograph. The sum of the non-beryllium metallic elements is substracted from 100 to give the percent beryllia composition.

4.5.2.3 Test Data - The same lot of beryllia was used throughout the program. Several analyses were made in the various stages and are recorded in Table VI. The slight variations may reflect the reproducibility of analysis as well as segregation of impurities.

4.5.3 Surface Roughness

4.5.3.1 Specification - The surface roughness of the working surface of the substrate shall not exceed 4.0 microinches center line average (CLA).

4.5.3.2 Test Method - Surface roughness is measured in accordance with ANSI B46.1-1962. A Clevite Surfanalyzer is used to give a graphical reading. The cutoff width is 0.030 in. and the stylus speed is 0.1 in./sec.

4.5.3.3 Test Results - Surface roughness was measured on five Type A parts and two input plates to dicing of Type A parts, 37 Type B substrates, and 37 Type C substrates. The results are collected in Table VII. The data show that the average surface finish increases in roughness as the size of the part increases. This probably

TABLE VI

COMPOSITION OF BERYLLIA SUBSTRATES

Impurity Element	% By Weight			
	First Article		Pilot Run Type A	
	Sample 1	Sample 2	Sample 1	Sample 2
Al	.0045	.0055	.0033	.0028
Fe	.0055	.0050	.0054	.0066
Mg	.0990	.1100	.1050	.1000
Si	.1850	.1500	.1700	.1900
ΣOthers	.0130	.0154	.0116	.0114
Total	.3070	.2859	.2953	.3108
% BeO	99.69	99.71	99.70	99.69

	Pilot Run Type B		Pilot Run Type C	
	Sample 1	Sample 2	Sample 1	Sample 2
Al	.0030	.0032	.0030	.0043
Fe	.0048	.0055	.0057	.0052
Mg	.1080	.1150	.1120	.1200
Si	.1800	.2000	.1900	.1700
ΣOthers	.0114	.0115	.0115	.0115
Total	.3072	.3352	.3222	.3110
% BeO	99.69	99.66	99.68	99.69

Overall Average % BeO = 99.69

TABLE VII

SURFACE FINISH OF POLISHED SUBSTRATES

Surface Finish, Working Surface, Microinches	Type A Substrates, Frequency	Type B Substrates, Frequency	Type C Substrates, Frequency
2.7	1		
2.8	2		1
2.9	1	3	2
3.0	2	4	1
3.1	1	4	0
3.2		8	1
3.3		6	3
3.4		6	4
3.5		3	7
3.6		3	14
3.7			2
3.8			2
		Weighted Average	
Microinches	2.9	3.2	3.5

reflects the movement of parts within the polishing ring. The type A plates were held rigidly in position by the double adhesive tape. The larger parts were restricted only by the thick diamond paste and the felt backing.

4.5.4 Surface Homogeneity

4.5.4.1 Specification - The substrate working surface shall be free of inclusions, scratches, cracks, pits, and blisters.

4.5.4.2 Test Method - The surface homogeneity examination is made in accordance with ASTM E165, Test Method B. The visible Turco Dy-Chek Penetrant WW-1 is used. Substrates are examined under 10 and 40 times magnification for defects.

4.5.4.3 Test Results - All 48 samples inspected were acceptable. The following criteria are used at Brush Wellman Inc. for rejection:

Blemish - maximum diameter of one blemish allowable is 0.020 in./part

Blister - maximum diameter of one blister allowable is 0.010 in./part

Burr, Fin, Flash - none allowed

Chip - Type A - 0.020 in. wide x 0.020 in. deep, maximum

Type B and C - 0.040 in. wide x 0.040 in. deep, maximum

Cracks and Laminations - Maximum 0.020 in. from edge into part

Inclusion - One inclusion > 0.015 in. diameter

Scratch - Within surface finish tolerance.

4.5.5 Thermal Shock

4.5.5.1 Specification - The substrate when subjected to one cycle of the test shall show no cracks, pits, blisters, pores, or change of appearance.

4.5.5.2 Test Method - The test substrate is placed on a preheated beryllia setter tile in a furnace at temperature and heated to 500°C ±10°C and maintained for 15 minutes. The setter tile and substrate are removed from the furnace, and the substrate is slid onto ceramic insulation at room temperature of 25°C ±1°C to cool for 10 minutes. The substrate is returned to the setter tile from which it is slid into a flask containing liquid nitrogen. After 10 minutes immersion in the liquid nitrogen, the substrate is returned to the ceramic insulation and allowed to warm to ambient temperature. The substrates are examined for defects according to ASTM D116 Test Method D as in paragraph 4.5.11.2.

4.5.5.3 Test Results - Four samples of each substrate type were submitted to the thermal cycle described. No damage resulted from the thermal shock cycle to any substrate.

4.5.6 Thermal Conductivity

4.5.6.1 Specification - The thermal conductivity value shall be no less than 0.61 g-cal/cm^2/cm/sec/°C at 25°C and no less than 0.45 g-cal/cm^2/cm/sec/°C at 100°C.

4.5.6.2 Test Method - Thermal conductivity was measured at 25°C and 100°C by Theta Industries Inc. by the ASTM C408 test procedures. Exceptions to the procedure are (1) the test specimen is 0.500 ±0.001 in. diameter and 0.750 ±0.001 in.

long and (2) the test specimen does not have any metalized surface. The measurements are made with Type E thermocouples and a Theta Industries Inc. conductivity measurement system, Model 8706.

4.5.6.3 Test Data - Six samples were measured during the course of the program, supplying the following:

Sample	Thermal Conductivity, g-cal/cm^2/cm/sec/°C At 25°C	At 100°C
Engineering	0.73	0.52
	0.74	0.50
First Article	0.85	0.45
	0.74	0.45
Pilot Run	0.79	0.48
	0.73	0.48

The special test specimens were made from the same lot of ready-to-press beryllia powder and were fired under the same thermal cycle conditions as the substrates. The specific gravity was 2.91 g/cc for both of the special test specimens used in the Pilot Run evaluation.

4.5.7 Dielectric Constant and Loss Tangent

4.5.7.1 Specification - The dielectric constant and loss tangent at testing temperatures of 25°C and 100°C shall be in accordance with the table below when the measurements have been made in accordance with ASTM D150-65 or ASTM 2520-66T.

Test Frequency	Dielectric Constant Minimum	Dielectric Constant Maximum	Loss Tangent Maximum At 25°C	Loss Tangent Maximum At 100°C
1 MHz	6.6	6.8	0.0003	0.0009
1 GHz	6.6	6.8	0.0003	0.0003
10 GHz	6.6	6.8	0.0003	0.0004

4.5.7.2 Test Method - A special test specimen is used at Coors Spectro-Chemical Laboratory. It is a 1.80 in. diameter disc of 0.09 in. thickness. ASTM D150-65 test procedure for the micrometer electrode technique is used for the 1 MHz measurements at 25°C and 100°C. The 1 GHz measurements at 25°C and 100°C use the same special test specimen in test procedure ASTM 2520-66T. The 10 GHz measurements are made at 25°C and 100°C according to test procedure ASTM 2520-66T, using a special test specimen 3.5 in. long and 0.25 in. diameter. Both special test specimens were made from the same blended lot of ready-to-press powder and were fired under the same thermal cycle conditions as the substrates.

4.5.7.3 Test Data - Sixteen measurements were made at the 1 MHz frequency and six were made for the 1 GHz as well as the 9.3 GHz frequency. The results are shown in Table VIII. Not all of the data meet the original SCS-472 specification or the contractually modified values listed in paragraph 4.5.7.1. Density of the special disc specimens was 2.91 g/cc for both specimens used in the Pilot Run evaluation. Density was 2.93 and 2.90 g/cc for the two special rod samples used in the Pilot Run evaluation.

4.5.8 Volume Resistivity

4.5.8.1 Specification - The volume resistivity of the substrate shall be no less than 10^{15} ohm-cm at 25°C and no less than 10^{13} ohm-cm at 100°C, as contractually modified from the SCS-472 specification.

TABLE VIII

DIELECTRIC CONSTANT AND LOSS TANGENT

Test Frequency	Dielectric Constant At 25°C	Dielectric Constant At 100°C	Loss Tangent At 25°C	Loss Tangent At 100°C
1 MHz	6.67	6.69	0.00012	0.00010
	6.68	6.67	0.00010	0.00013
	6.72	6.75	0.00006	0.00012
	6.69	6.70	0.00005	0.00011
	6.72	6.74	0.00006	0.00011
	6.71	6.73	0.00005	0.00008
	6.69	6.71	0.00007	0.00013
	6.70	6.74	0.00006	0.00010
	6.60	6.64	0.00010	0.00009
	6.64	6.64	0.00012	0.00012
	6.59	6.68	0.00010	0.00012
	6.65	6.73	0.00012	0.00009
	6.67	6.75	0.00007	0.00007
	6.64	6.74	0.00008	0.00008
	6.55	6.55	0.00016	0.00019
	6.59	6.59	0.00011	0.00017
Average	6.66	6.69	0.00009	0.00010
1 GHz	6.72	6.72	0.00023	0.00034
	6.72	6.72	0.00023	0.00051
	6.75	6.73	0.00030	0.00014
	6.74	6.79	0.00035	0.00024
	6.75	6.81	0.00007	0.00026
	6.75	6.81	0.00006	0.00023
Average	6.74	6.76	0.00021	0.00029
9.3 GHz	6.77	6.77	0.00031	0.00037
	6.77	6.77	0.00031	0.00038
	6.76	6.77	0.00030	0.00026
	6.76	6.77	0.00024	0.00041
	6.77	6.77	0.00007	0.00047
	6.77	6.77	0.00008	0.00047
Average	6.77	6.77	0.00021	0.00039

4.5.8.2 Test Method - The volume resistivity measurements were made at Coors Spectro-Chemical Laboratory using ASTM D257 and D1829 (Procedure A). The special test specimen is the same 1.80 in. diameter disc of 0.09 in. thickness used in paragraph 4.5.7.2 for dielectric constant and loss tangent. The measurements were made with a General Radio Company megohm bridge and a shielded electrode furnace assembly.

4.5.8.3 Test Data - Six samples were measured during the program, supplying the following:

Sample	Volume Resistivity ohm-cm At 25°C	At 100°C
Engineering	2.0×10^{16}	1.4×10^{13}
	3.0×10^{16}	2.3×10^{13}
First Article	3.2×10^{16}	5.0×10^{13}
	2.2×10^{16}	3.5×10^{13}
Pilot Run	1.2×10^{17}	2.8×10^{13}
	1.3×10^{17}	2.8×10^{13}

Contractual modification of the 10^{14} ohm-cm minimum at 100°C in the SCS-472 specification to 10^{13} ohm-cm was needed after Engineering Sample testing. This was confirmed by the values reported in the First Article and Pilot Run phases of the program.

4.5.9 Warpage

4.5.9.1 Specification - The warpage of the substrate shall not exceed 0.003 in./in.

4.5.9.2 Test Method - The thickness is measured as in paragraph 4.5.1.3 and the lowest reading is recorded. The overall thickness is measured with a dial gage stand from Mitutoyo Manufacturing Company. This instrument has

parallel plates with a diameter greater than the substrate's diagonal or largest dimension. The difference between the two measurements is the total warpage. Total warpage is divided by the longest dimension of the substrate to give unit warpage.

4.5.9.3 Test Data - For each of the three substrate types, 48 samples were measured for warpage. The distribution of the values is given in Table IX. For the small area Type A parts, the warpage reported is more likely the difference in thickness between two instruments used for the same measurement.

4.5.10 Specific Gravity

4.5.10.1 Specification - The specific gravity shall be no less than 2.85.

4.5.10.2 Test Method - ASTM C20-70 is the procedure used to determine the specific gravity. Type A substrates have such small mass that the difference between weighing in air and in water is less than the accuracy of the analytical balance. The 1 in. x 1 in. x 0.010 in. substrate from which Type A substrates had been diced was monitored for compliance with the specific gravity requirement.

4.5.10.3 Test Data - Three input plates to Type A dicing, 48 polished Type B substrates, and 48 polished Type C substrates were measured, giving the frequency distributions collected in Table X. The specific gravity values are considerably above the specification minimum and slightly above typical values; this might explain the above typical values of thermal conductivity reported in paragraph 4.5.6.3.

TABLE IX

WARPAGE IN POLISHED SUBSTRATES

Unit Value, in./in.	Type A, Frequency	Type B, Frequency	Type C, Frequency
0.00000	28	0	0
0.00005	0	0	4
0.00010	0	19	9
0.00020	0	5	11
0.00030	0	6	8
0.00040	0	0	2
0.00050	5	6	5
0.00060	0	0	0
0.00080	0	0	6
0.00090	0	0	0
0.00100	8	8	3
0.00130	0	1	3
0.00140	0	1	0
0.00150	0	0	1
0.00200	7	2	0

TABLE X

SPECIFIC GRAVITY OF SUBSTRATES

Specific Gravity	Frequency (Number of Observations)		
	Type A	Type B	Type C
2.88	1	0	0
2.89	1	0	3
2.90	0	4	41
2.91	1	33	2
2.92	0	5	2
2.93	0	2	0
2.94	0	3	0
2.96	0	1	0

4.5.11 Porosity

4.5.11.1 Specification - The substrate shall not show evidence of dye penetration.

4.5.11.2 Test Method - Method D in ASTM D116 is the procedure for determining porosity. The substrates are immersed in the water washable dye penetrant tank for 10 min. After draining, the excess dye is removed by washing in a rinse tank. The parts are dried, then examined under 40 times magnification for dye retention in pores.

4.5.11.3 Test Results - Brush Wellman Inc. inspects for porosity and rejects for any pit, pock, pore, hole, void, or porous area (surface dimension) in excess of 0.020 in. diameter. The 48 specimens of Types A, B, and C inspected met these requirements.

4.5.12 Flexural Strength

4.5.12.1 Specification - The substrate shall show a flexural strength greater than 30,000 psi.

4.5.12.2 Test Method

4.5.12.2.1 Specimen Preparation - Special test specimens are made from the same lot of ready-to-press beryllia powder and are fired under the same thermal cycle conditions as the substrate. They are pressed and fired individually to the approximate dimensions of 0.210 in. $\underline{b}$ x 0.210 in. $\underline{d}$ x 1.85 in. $\underline{l}$. The specimens are machined on the Blanchard grinder with #100 grit metal-bonded wheel to the approximate dimensions of 0.185 in. $\underline{b}$ x 0.185 in. $\underline{d}$ x 1.85 in. $\underline{l}$. Finishing is done

on a surface grinder with a #320 grit vitrified-bonded wheel. Finished specimens meet the following requirements:

b & d = 0.175 $\pm$0.001 in., flat and parallel to 0.0005 in.

l = 1.85 in.

Surface finish is 20-30 microinch. Specimens are dye-penetrant inspected for visual defects as in paragraph 4.5.11.2.

4.5.12.2.2 Breaking Procedure - A Dillon Universal testing machine, Model M, with a 300 lb. load capacity is used. The specimen is loaded onto two supporting knife-edges 1.75 in. apart. A third knife-edge is centered between and opposing the other two, providing a three point loading apparatus. All knife-edges have a 0.125 in. radius. Load is applied at the rate of $\sim$ 28 lbs. per minute. The cross-head speed is 0.062 in. per minute. Six specimens are broken for a standard test.

4.5.12.2.3 Calculation of Modulus of Rupture

The modulus of rupture is calculated according to the equation

$$M \text{ of } R = 3Pl/2bd^2$$

where

M of R - modulus of rupture

P = breaking load in lbs.

l = span in inches between supporting knife-edges

b = breadth of specimen in inches

d = depth in inches (dimension across which load is applied)

The average value for the six specimens is reported.

4.5.12.3 Test Data - Ten specimens were broken in the Engineering Samples, First Article, and Pilot Run phases of the program. The average value for each group of specimens was:

Engineering Samples	35,843 psi
First Article	39,355 psi
Pilot Run	34,998 psi

Only one of the 30 specimens broken was below the 30,000 psi specification. Its low value of 24,649 psi leads to the suspicion that the specimen had a machining flaw. Density of the special test specimens-2.89 g/cc for six of the specimens and 2.90 g/cc for the other 24 specimens.

4.6 Specifications Review

4.6.1 Dimensions - No change from the dimensions specification of the substrates in Table I of SCS-472 is required.

4.6.2 Substrate Composition - No change from the requirement of 99.5% beryllia or above in SCS-472 is required.

4.6.3 Surface Roughness - The requirement of SCS-472 that the surface roughness of the working surface of the substrate shall not exceed 4.0 microinches center line average (CLA) and a primary objective of this program remain acceptable without change.

4.6.4 Surface Homogeneity - The specification as stated in SCS-472 is accepted, however, to quantify this specification the Brush Wellman Inc. criteria in paragraph 4.5.4.3 are suggested for converting

a subjective to an objective specification.

4.6.5 Thermal Shock - The requirements of SCS-472 are accepted.

4.6.6 Thermal Conductivity - Requirements of SCS-472 are acceptable for the 25°C testing condition, although the minimum requirement might be increased to 0.65 g-cal/cm^2/cm/sec/°C at 25°C as a result of this program. The specification of SCS-472 at 100°C should be lowered, if the specific gravity requirement of paragraph 4.5.10.1 remains at a 2.85 minimum. These two properties are closely interrelated.

4.6.7 Dielectric Constant and Loss Tangent - As a result of the data obtained in the Engineering Samples, First Article, and Pilot Run phases of the program, the dielectric constant and loss tangent at testing temperatures of 25°C and 100°C should be in accordance with:

Test Frequency	Dielectric Constant (Minimum)	(Maximum)	Loss Tangent (Maximum)
1 MHz	6.6	6.8	0.0003
1 GHz	6.6	6.8	0.0004
10 GHz	6.6	6.8	0.0005

No change from the original SCS-472 specification for loss tangent is required. The dielectric constant minimum and maximum values should be increased as supported by the data reported in paragraph 4.5.7.3 and Table VIII. In reality, there should be no maximum limitation.

4.6.8 Volume Resistivity - The SCS-472 specification of a minimum value of 10^{15} ohm-cm at 25°C is accepted. The contractual modification that the volume resistivity at 100°C should be no less than 10^{13} ohm-cm is sustained.

4.6.9 Warpage - The requirements of Specification SCS-472 are accepted.

4.6.10 Specific Gravity - The minimum specific gravity of 2.85 in the SCS-472 specification should remain unchanged.

4.6.11 Porosity - Specification SCS-472 states that the substrate shall not show evidence of dye penetration. To make this an objective specification, the Brush Wellman Inc. inspection requirement is recommended, i.e., no pit, pock, pore, hole, void, or porous area (surface dimension) shall be in excess of 0.020 inch diameter.

4.6.12 Flexural Strength - The minimum value of 30,000 psi in specification SCS-472 is accepted.

4.6.13 List of Test Apparatus - The list of test apparatus is found in Table XI.

4.7 Conclusions

It has been demonstrated that a two step progressive polishing process can be used to produce a super fine finish working surface on beryllia substrates with surface roughness reduced to less than four microinches center line average. Commercially available Thermalox 995 or equivalent input plates can be used. Installed manufacturing scale equipment was modified only to the extent of adding two 24 inch diameter spiral groove polishing wheels to accomplish polishing with 6 micron and 3 micron diamond paste in the First Article and Pilot Run phases of the program. Polishing of larger plates from which the 0.080 in. x 0.110 in. Type A parts were produced by dicing was a substantial cost saving step. Production rates of 1000 polished substrates per five day week in one shift were demonstrated for each of the three substrate sizes.

TABLE XI

LIST OF APPARATUS

Item No.	Equipment	Manufacturer	Model No.	Serial or Inventory No.	Date or Frequency of Calibration
1	Micrometer	Brown & Sharpe	-	QCC-12	1/17/76
2	Micrometer	Lufkin	-	1943	2/10/76
3	Dial Gage Stand	Mitutoyo	7001/ 7003	2918	1/5/76
4	Balance	Mettler	H-18	446510	5/21/75
5	Universal Testing Machine	Dillon	M	771	7/1/75
6	Micrometer	Starrett	230	CL-1	1/6/76
7	Micrometer	Starrett	2	CL-2	1/6/76
8	Grinder	Blanchard	11	3179	N/A*
9	Grinder	Brown & Sharpe	824	523-824-596	N/A
10	Spectrograph	Jarrell-Ash	JA-7101	57752	N/A
11	Microphotometer	Jarrell-Ash	2100	25-5945	Each use
12	Calculating Board	Jarrell-Ash	3001	-	N/A
13	Photoprocessor	Jarrell-Ash	3410	25-5943	N/A
14	Furnace	Lindberg	51123	20652	N/A
15	Surfanalyzer	Clevite	21-1330-20	409	Each use
16	Stereo Magnifier	Olympus	sZ	208425	N/A
17	Q Meter	Boonton Radio	260A	6321	3/4/76
18	Inductor	Boonton Radio	103A	21	N/A
19	Q Standard	Boonton Radio	513A	1552	24 mo.
20	Oscilloscope	Tektronix	503	1476	N/A
21	Frequency Counter	Hewlett-Packard	52466	816-01755	N/A
22	Null Detector	General Radio	1232A	1601	N/A

Item No.	Equipment	Manufacturer	Model No.	Serial or Inventory No.	Date or Frequency of Calibration
23	Klystron Power Supply	Hewlett-Packard	716B	6957	N/A
24	Variable Attenuator	Hewlett-Packard	X-382A	10694	24 mo.
25	Sample Cavity	Bureau of Standards	-	G-8	N/A
26	Sample Cavity	Bureau of Standards	-	G-1	N/A
27	Power Meter	Hewlett-Packard	432A	1141A11975	24 mo.
28	Megohm Bridge	General Radio	1644-A	181	24 mo.
29	Data Logger	Digitec	1268	-	N/A
30	Conductivity Measurement System	Theta Industries	8706	-	4/15/76
31	DC Power Supply	Sorenson	SRL 105	-	N/A
32	Thermocouples	Omega Engineering	E	-	4/15/76

*N/A = Not Applicable

Comparison of the Pilot Run demonstration with the state-of-the-art at the start of the program is difficult. At the end of the first quarter of the program, Type B substrates could probably be made at a rate of one part/man-hour with a yield near 40-50% using a laboratory-scale Speedfam 12 polisher. In the Pilot Run, 22 parts/man-hour were produced on a Speedfam 24 polisher at an 85% yield. The same technology on the Speedfam 12 polisher is estimated to yield over six Type B substrates/man-hour for a six-fold increase or better as a result of the program. Input beryllia plate costs for products and losses are a comparatively small fraction of the final cost of super fine finish beryllia substrates at a 1000 part/week level of production. It is estimated that production costs were reduced a factor of five through the program.

4.8 Additional Work Requirements

The evolved polishing process is highly labor intensive, so additional work should be concentrated in this area. With appropriate market requirements, incentive for such activity would be provided by the profit motive. Obvious areas for improvement include:

(1) Improved method of attaching Type A input plates to the weights of the Speedfam 12 polisher; success in this direction is promised in limited tests of the Crystalbond 555 adhesive.

(2) Automated sorting of input plates by thickness into 0.001 inch increments to improve the rate of full coverage on all parts in a given polishing ring.

(3) Automated cleaning of polished substrates by counter-current washing in an ultrasonic cleaning station equipped for unattended drying.

(4) Identification of a replacement for the DeKhotinsky cement used to mount parts on the master saw board. Removal of the residual cement from Type A parts contributes about 40% to the cost of producing these parts.

SECTION V

PROCESS MANUAL FOR PEM FOR SUPER FINE FINISH BERYLLIA

5.1 Incoming Inspection of Beryllia Plates

5.1.1 Dimensions - Dimensions of the input plates shall be as specified in Table II, which is repeated below:

TABLE II

DIMENSIONS OF INPUT PLATES

All dimensions in inches

Type	Length	Width	Thickness
A	1.0±0.005	1.0±0.005	0.013±0.002
B	1.0±0.005	1.0±0.005	0.029±0.002
C	2.0±0.010	2.0±0.010	0.044±0.003

5.1.2 Thickness Verification and Sorting - Measure the thickness of all input plates. Reject those plates which exceed the tolerances stated in Table II. Sort acceptable Type B and C plates according to thickness, grouping into one mil increments.

5.1.3 Visual Inspection - While measuring thickness as in paragraph 5.1.2, visually examine input plates for cracks and surface defects. Reject unacceptable plates.

5.2 Preparation of Supplies

5.2.1 Lubricant - Prepare lubricant for the polishing process by mixing one gallon of reagent-grade ethylene glycol with five gallons of distilled or deionized water in a clean container.

5.2.2 Diamond Paste - Inspect all diamond polishing compound to ensure that it is water-base type. Verify that adequate supplies of 6 micron and 3 micron diamond paste grades are available. Confirm that the

numerical grade of the paste corresponds to that on the polishing wheel to be used.

5.3 Procedure for Six Micron Diamond Polishing of Type A Plates

5.3.1 Preparation of Equipment - Thoroughly clean the Speedfam 12 polishing machine, retaining rings, and pressure heads. Install the 6 micron diamond spiral groove polishing wheel, identified by the number 6 stamped on the wheel. Select and confirm that a tube of 6 micron diamond paste is the only type on the polishing table.

5.3.2 Loading of Polisher - Identify the non-working surface of twenty Type A input plates by marking with a felt ink pen. Attach five input plates to each of the four dry pressure heads by means of double-sided adhesive tape. Distribute the parts near the outer circumference of the pressure head with approximately uniform separation. If the polishing wheel has not been used within the last hour, generously wet the surface of the wheel with the ethylene glycol lubricant. Allow the lubricant to penetrate any dried paste on the wheel. Position the four pressure heads within their retaining rings and locate against the retaining rollers. Apply about one-half gram of 6 micron diamond paste across the face of the polishing wheel. Apply ethylene glycol lubricant by spraying from a wash bottle.

5.3.3 Operation of Polisher - Set timer for 10 minutes and start the machine. During all polishing runs, apply additional lubricant to maintain a smooth, free-running polishing operation. A foamy condition occurs when excessive amounts of lubricant have been added. After 10 minutes of polishing, inspect parts for full

coverage of the surface by the polishing action. If coverage is complete, remove parts and measure thickness for in-process inspection. If the thickness is 0.012 in. or less, proceed to cleaning operation. This is generally the case. If polishing coverage is incomplete or the thickness remains greater than 0.012 in., repeat the steps in paragraphs 5.3.2 and 5.3.3 until the coverage and thickness requirements are met.

5.3.4 Cleaning of Polished Plates - With the completion of a polishing run, remove the parts and immediately immerse them in a soapy water solution, clean with a light scrubbing action in the water, rinse with distilled or deionized water, and dry. Visually inspect, reject any broken parts, but retain those with corner breakage only.

5.3.5 Post-Polishing Procedures - If additional 6 micron diamond polishing runs are scheduled, protect the machine from contamination with dirt or foreign matter by covering with a plastic bag between runs. When the 6 micron diamond polishing schedule has been completed, remove the polishing wheel, clean by washing only, and transfer to a clean plastic bag. Seal the bag and return the wheel to storage. Clean the pressure heads and retaining rings in soapy water, rinse, and thoroughly dry with forced hot air. Thoroughly clean the polishing machine and table. Cover the machine with a plastic bag, if continued usage of the polisher is not scheduled.

5.4 Procedure for Three Micron Diamond Polishing of Type A Plates

5.4.1 Preparation of Equipment - If the procedure of paragraph 5.3.5 is always followed, the polisher is always prepared to receive a polishing wheel designated for a different grade of polishing

compound. If not, repeat the machine cleaning steps of paragraph 5.3.5. Install the 3 micron diamond spiral groove polishing wheel, identified by the number 3 stamped on the wheel. Select and confirm that a tube of 3 micron diamond paste is the only type on the polishing table. Return any other polishing compounds to storage.

5.4.2 Loading of Polisher - Attach five polished parts processed through paragraph 5.3.4 to each of the four pressure heads with double-sided adhesive tape, keeping the unmarked working surface toward the polishing wheel. If the polishing wheel has not been used within the last hour, generously wet the surface of the wheel with the ethylene glycol lubricant. Allow the lubricant to penetrate any dried paste on the wheel. Position the four pressure heads within their retaining rings and locate against the retaining rollers. Apply about one-half gram of 3 micron diamond paste across the face of the polishing wheel.

5.4.3 Operation of Polisher - Set timer for 10 minutes and start the machine. During all polishing runs, apply additional lubricant by spraying from a wash bottle to maintain a smooth, free-running polishing operation. A foamy condition occurs when excessive amounts of lubricant have been added. After 10 minutes of polishing, inspect the parts for full coverage of the surface by polishing action. Full coverage is generally achieved during this period. If coverage is not complete, return the pressure heads to the retaining rings. Reset the time for 10 minutes, start the machine, and continue the polishing as before. After polishing for 10 minutes, inspect the parts again for full coverage by polishing. Repeat, as needed.

5.4.4 Cleaning of Polished Plates - With the completion of a polishing run, remove the parts and immediately immerse them in a soapy water solution, clean with a light scrubbing action in the water, rinse with distilled or deionized water, and dry. Visually inspect for cracked or broken parts and newly exposed inclusions. Parts with broken corners only are acceptable.

5.4.5 Post-Polishing Procedures - If additional 3 micron diamond polishing runs are scheduled, protect the machine from contamination with dirt or foreign matter by covering with a plastic bag between runs. When the 3 micron diamond polishing schedule has been completed, remove the polishing wheel, clean by washing only, and transfer to a clean plastic bag. Seal the bag and return the wheel to storage. Clean the pressure heads and retaining rings in soapy water, rinse, and thoroughly dry with forced hot air. Thoroughly clean the polishing machine and table. Cover the machine with a plastic bag if continued usage of the polisher is not scheduled.

5.5 Dicing of Type A Substrates

5.5.1 Mounting Polished Plates on Master Saw Board - The acceptable plates processed through paragraph 5.4.4 are mounted on a master saw board, polished surface of the plates being up. The board and plates are placed on a hot plate. When the set-up is at temperature, DeKhotinsky cement is applied and melted on the parts. After carefully aligning the beryllia plates to minimize dicing losses, the hot plate is turned off so the cement can cool and solidify.

5.5.2 Sawing of Mounted Plates - The master saw board and polished plates are clamped in the holding fixture of the Do-All grinder. The grinder is equipped with a seven inch diameter diamond-edge

saw blade of 0.020 inch thickness. The machine has been preset to cut .110 in. wide strips in a step and repeat pattern. The machine speed is set at 3200 rpm and the feed rate is set at 2.5 in./minute. The .110 in. wide strips are sawed. The master board is rotated 90°, the machine is reset to cut .080 in. strips, and sawing proceeds in a step and repeat pattern.

5.5.3 Recovery of Type A Substrates - After dicing, the master board is heated on a hot plate to melt the DeKhotinsky cement and release the small parts. Residual cement is removed from the substrates by soaking in warm Jaysol or similar alcohol-base cleaner.

5.6 Procedure for Six Micron Diamond Polishing of Type B and C Plates

5.6.1 Preparation of Equipment - Thoroughly clean the Speedfam 24 polishing machine, retaining rings, and pressure heads. Install the 24 in. diameter, 6 micron diamond spiral groove polishing wheel, identified by the number 6 stamped on the wheel. Select and confirm that a tube of 6 micron diamond paste is the only type on the polishing table.

5.6.2 Loading of Polisher - Load 52 of the Type B input plates of the same thickness group or 12 of the Type C input plates of the same thickness groups into each of the four retaining rings. Type B and Type C plates cannot be co-mingled in a retaining ring, but Type B-containing rings and Type C-containing rings can be simultaneously mounted in the polisher. If the polishing wheel has not been used within the last hour, generously wet the surface of the wheel with the ethylene glycol lubricant. Allow the lubricant to penetrate any dried paste areas on the wheel. Place the felt

backing disc over the parts. Lower the pressure head inside the retaining ring against the felt disc and parts. Apply about one gram of 6 micron diamond paste across the face of the wheel. Generously apply lubricant by spraying from a wash bottle.

5.6.3 Operation of Polisher

5.6.3.1 Flattening Step - Set the timer for ten minutes with the air pressure gauge at zero. Start the machine and immediately increase the air pressure to four psi gauge, which provides a pressure of one psi gauge per part. Continuously apply lubricant during all Speedfam 24 runs to maintain free-running polishing action. After ten minutes, raise the pressure heads and remove the felt pads. Invert or flip the parts in each retaining ring. Inspect the condition of all felt pads, replacing any that are damaged. Insert the felt pads and lower the pressure heads. Apply about one gram of 6 micron diamond paste and lubricate generously.

5.6.3.2 Polishing Step - Set the timer for twelve minutes with the air pressure gauge at zero. Start the machine and immediately increase the air pressure to four psi gauge. Continuously add lubricant to maintain free-running polishing action. At completion of the twelve minutes, raise the heads and remove the felt discs. Inspect the parts for full coverage of the surface by polishing action. Inspect a few of the parts for thickness to estimate the rate of material removal. If full coverage has not been achieved, or if the thickness exceeds the maximum

permissible, redistribute the parts and resume polishing for the time estimated to meet the coverage and thickness requirements.

5.6.4 Cleaning of Polished Plates - On completion of the 6 micron diamond polishing run, remove the parts and immediately immerse them in a soapy water solution, clean with a light scrubbing action, rinse with distilled or deionized water, and dry. Visually inspect for cracked or broken plates and for newly exposed inclusions; reject defective plates.

5.6.5 Post-Polishing Procedures - If additional 6 micron diamond polishing runs are scheduled, protect the machine from contamination with dirt or foreign matter by covering with a plastic bag between runs. When the 6 micron diamond polishing schedule has been completed, remove the polishing wheel, clean by washing only, and transfer to a clean plastic bag. Seal the bag and return the wheel to storage. Clean the retaining rings and felt discs in soapy water, as well as the pressure heads, rinse, and thoroughly dry with forced hot air. Thoroughly clean the polishing machine and table. Cover the machine with a plastic bag, if continued usage of the polisher is not scheduled.

5.7 Procedure for Three Micron Diamond Polishing of Type B and C Plates

5.7.1 Preparation of Equipment - If the procedure of paragraph 5.6.5 is always followed, the polisher is prepared to receive a polishing wheel designated for a different grade of polishing compound. If not, repeat the machine cleaning steps of paragraph 5.6.5. Install the 3 micron diamond, 24 in. diameter, spiral groove

polishing wheel, identified by the number 3 stamped on the wheel. Select and confirm that a tube of 3 micron diamond paste is the only type on the polishing table. Return any other polishing compounds to storage.

5.7.2 <u>Loading of Polisher</u> - Load 52 of the Type B or 12 of the Type C plates acceptable after processing through paragraph 5.6.4 from the same original thickness groups into each of the four retaining rings with the fully polished side facing the polishing wheel. Type B and Type C plates cannot be co-mingled in a retaining ring, but Type B-containing rings and Type C-containing rings can be simultaneously mounted in the polisher. Mark the upper side of all parts with a waterproof felt marking pen. If the polishing wheel has not been used within the last hour, generously wet the surface of the wheel with the ethylene glycol lubricant. Allow the lubricant to penetrate any dried paste areas on the wheel. Place the felt backing disc over the parts. Lower the pressure heads inside the retaining ring against the felt disc and parts. Apply about one gram of 3 micron diamond paste across the face of the wheel. Generously apply lubricant by spraying from a wash bottle.

5.7.3 <u>Operation of Polisher</u> - Set timer for 10 minutes with the air pressure gauge at zero. Start the machine and immediately increase the air pressure to four psi gauge. Continuously apply lubricant by spraying during the run to maintain free-running polishing action. At the conclusion of the run, raise the pressure heads and the felt discs.

5.7.4 Cleaning of Polished Substrates - Remove the polished substrates from the retaining rings and immediately immerse them in a soapy water solution, clean with a light scrubbing action in the water, rinse with distilled or deionized water, and dry. Visually inspect for full polishing coverage; return unacceptable parts for additional polishing. Reject cracked or broken parts and any with newly exposed inclusions.

5.7.5 Post-Polishing Procedure - If additional 3 micron diamond polishing runs are scheduled, protect the machine from contamination with dirt or foreign matter by covering with a plastic bag between runs. When the 3 micron diamond polishing schedule has been completed, remove the polishing wheel, clean by washing only, and transfer to a clean plastic bag. Seal the bag and return the wheel to storage. Clean the retaining rings, felt discs, and pressure heads in soapy water, rinse, and thoroughly dry with forced hot air. Thoroughly clean the polishing machine and table. Cover the machine with a plastic bag, if continued usage of the polisher is not scheduled.

SECTION VI

IN-PROCESS QUALITY ASSURANCE

6.1 Incoming Inspection

6.1.1 Dimensions - 100% inspection of the thickness dimension of the input beryllia plates is made. Micrometer readings are taken at the four corners of each plate with the 0 -1 in. range Brown and Sharpe micrometer. Plates exceeding the thickness maximum of Table II are accumulated until enough are available to fill a polishing ring. Plates with thickness below the minimum of Table II are rejected. Acceptable Type B and Type C plates are sorted into groups of one mil thickness increments. If the supplier has properly inspected the shipment, yields through this inspection should approach 100%.

6.1.2 Visual Inspection - While checking the dimensions, the beryllia plates are visually examined for cracks and surface defects. Except for damage during shipping, yields through this inspection should approach 100%.

6.2 In-process Inspection During Type A Substrate Production - The thickness requirement of the Type A input plates is obtained by machining the surfaces to reduce the as-fired thickness. Consequently, the surfaces are flat and parallel.

6.2.1 Six Micron Diamond Polishing - After ten minutes of polishing, the plates are inspected for complete coverage of the surface by the polishing action. If coverage is complete, the plates are removed. The thickness is measured at the corners of the plate with the 0 - 1 in. range Brown and Sharpe micrometer. This

inspection determines whether additional polishing is required to meet the final thickness specification. Visually examine for broken plates which are rejected. Plates with corners only lost by breakage are retained. Those with broken corners represent a final loss of about 3% in the dicing operation.

6.2.2 Three Micron Diamond Polishing - After 10 minutes of polishing, the parts are visually inspected for full coverage of the surface by the polishing action. If coverage is not complete, polishing is continued. This inspection does not reject any substrates.

6.2.3 Dicing - The first and last cut of the saw through the polished plate eliminates bowed or hour-glassed material from the edges of the plate. During recovery of the diced substrates, the operator must be observant that this sub-dimensional material is rejected.

6.3 In-process Inspection During Type B and Type C Substrate Production

6.3.1 Six Micron Diamond Polishing - Following the first ten minutes of polishing, the plates are inverted or flipped. During this operation, any cracked or chipped plates are removed and rejected. The felt discs are inspected for damage. After twelve minutes polishing of the working surface of the substrates, inspection is made for full coverage of the surface by the polishing action. A few of the parts are measured for thickness to give an estimate of the rate of material removal. This inspection determines whether further polishing is necessary. Yield is not affected, except for about 5% loss as cracked or chipped plates through collision in the retaining rings.

6.3.2 Three Micron Diamond Polishing - Following the ten minute polishing, the substrates are inspected for full surface coverage by the polishing action. If coverage is incomplete, polishing is continued for another period. During cleaning of the substrates, visual inspection is 100% to detect cracks or surface defects in the polished substrates. About 5% loss of substrates occurs during the final polishing step.

SECTION VII

IDENTIFICATION OF PERSONNEL

The following is a listing of Engineering personnel who contributed efforts to the program throughout its duration:

Glenn H. Rees, Senior Research Engineer, is a 1959 graduate of Pennsylvania State University with a B.S. degree in Ceramic Technology. Following graduation, he worked as a Development Engineer on chrome oxide and magnesite refractories with E. J. Lavino Refractories Company. From 1963 to 1966, Mr. Rees was Research Engineer for Universal Dental Company studying and developing crystallizable glasses and high-alumina porcelains. In 1966, he joined the Microelectronics Division of Philco-Ford Corporation as Process Engineer in their Circuit Module Group. There he worked with thick-film conductive and resistive materials as well as fabrication processing of multichip circuits. In 1968, Mr. Rees became Operations Manager of the Power Hybrid Group of The Silicon Transistor Corporation. In this assignment, he was responsible for the engineering and manufacture of hybrid circuits. In 1970 he joined Brush Wellman Inc. as Senior Research Engineer in the Product Development and Research Division to establish a Microelectronics Materials Laboratory. Work has been performed on the development of beryllia components having dielectric and conductive thick-film coatings for microcircuit package components. Studies have included the study of the effect of beryllia surfaces and crystal characteristics on metalization properties as a technical service contribution through the Ceramic Sales Department to promote technical liaison with beryllia substrate users. A recent assignment is the establishment of laboratory facilities for thin-film coating studies by vaporization-condensation and sputtering techniques in preparation for a long range fundamental study of these processes.

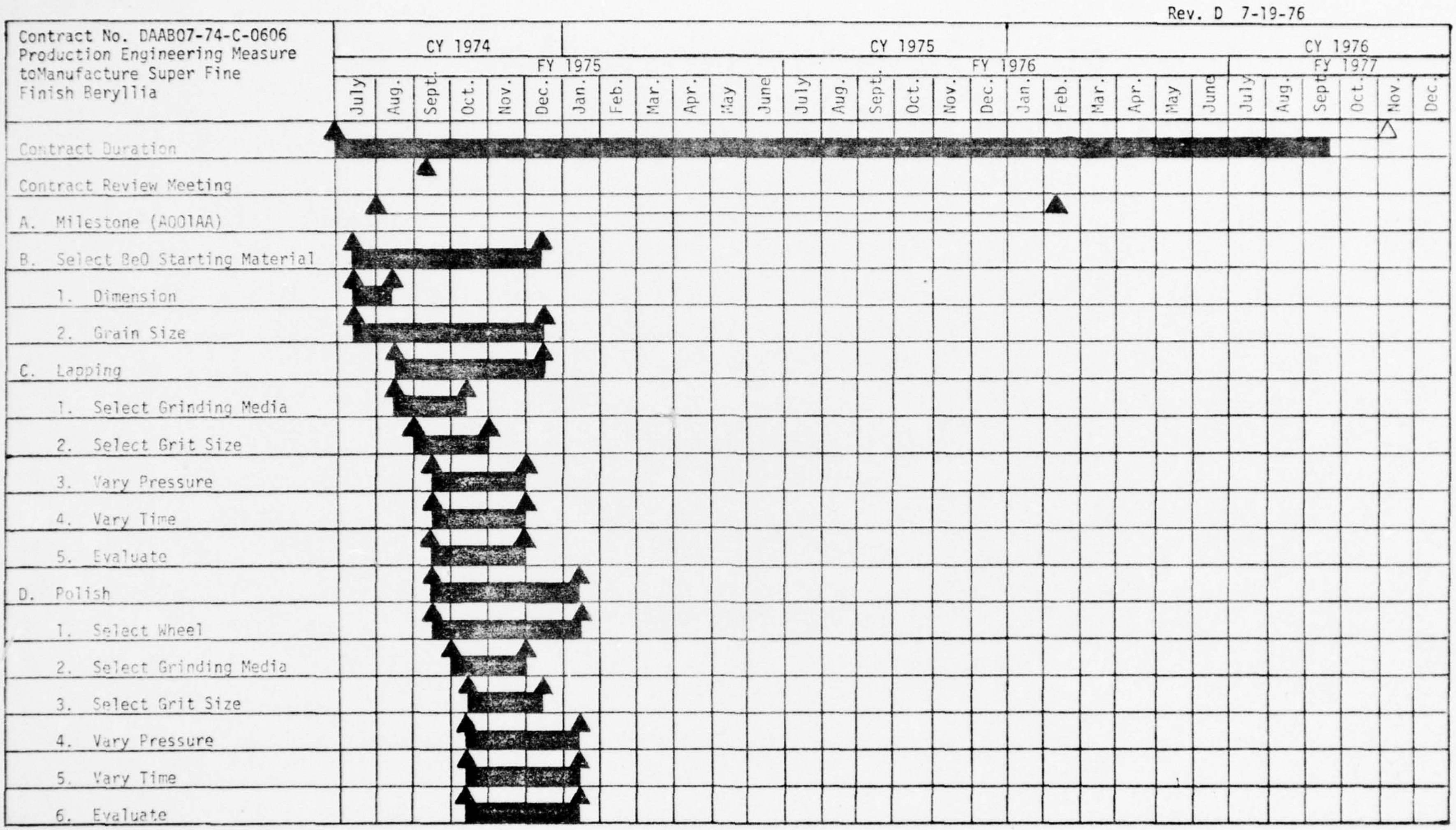
Eugene D. Nieset, PD&R technician, graduated from St. Joseph High School, Fremont, Ohio, 1963. He attended General Motors Institute for one year, majoring in Mechanical Engineering. He later attended the University of Toledo for one year, also majoring in Mechanical Engineering. He joined Brush Wellman Inc. in 1968 as an Analytical Assistant in the Spectrographic Laboratory. In 1972 he transferred to the Product Development and Research Department as a technician in the Electronic Materials Section. There he is involved in various technical aspects of ceramic materials fabrication, coating and joining techniques, and general electronic applications of beryllia ceramics.

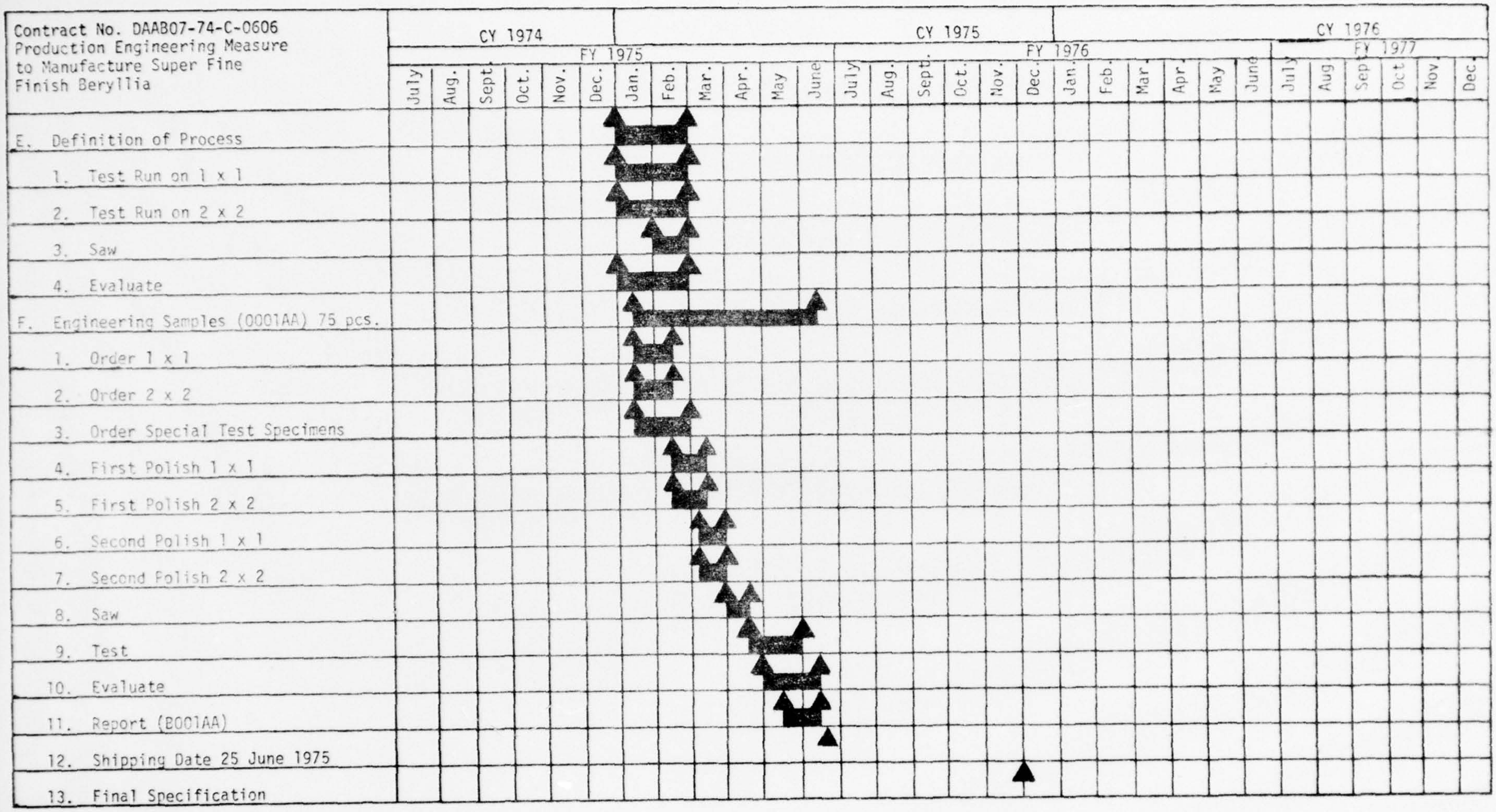
Dr. Kenneth A. Walsh, Associate Director, Corporate Technology, was graduated from Yankton College in 1942 with a B.A. degree. His graduate studies were interrupted in 1943, when he started work on the Manhattan Project at Ames, followed by a transfer to Los Alamos from 1944 to 1946. His early work concerned the chemistry of uranium and plutonium. Following World War II, he resumed his graduate studies doing research on beryllium and zirconium for which he received the Ph.D. degree in physical chemistry at Iowa State University in 1950. Following a year as Assistant Professor at Iowa State University, Dr. Walsh returned to Los Alamos Scientific Laboratory to work on solvent extraction processes, the pyrometallurgy of nuclear fuels, and the advanced chemistry of plutonium and americium from 1951 to 1957. In the 1957-1960 period he was Supervisor of Inorganic Chemical Research and Acting Manager of the Florida Experiment Station of International Minerals and Chemical Corporation. He joined Brush Wellman Inc. in 1960 as a Research Manager whose duties were gradually expanded from extractive metallurgy to chemical metallurgy to chemical metallurgy and ceramics. Administrative assignments in the Product Development and Research Division include technical support of the Elmore Ceramics Manufacturing and Sales

Departments, systems engineering approaches at Sawyer Research Products Inc., and recent entries into Corporate thin-film metalization and hydrothermal research programs.

Total efforts directed toward completion of the contract are distributed as follows:

	Man-Hours
Nieset, E.	722
Rees, G.	728
Walsh, K.	570
Manufacturing Personnel	262
Grand Total	2282


Rev. D 7-19-76
Contract No. DAAB07-74-C-0606
Production Engineering Measure
toManufacture Super Fine
Finish Beryllia
CY 1974
CY 1975
CY 1976
FY 1975
FY 1976
FY 1977
July
Aug.
Sept.
Oct.
Nov.
Dec.
Jan.
Feb.
Mar.
Apr.
May
June
Contract Duration
Contract Review Meeting
A. Milestone (A001AA)
B. Select BeO Starting Material
1. Dimension
2. Grain Size
C. Lapping
1. Select Grinding Media
2. Select Grit Size
3. Vary Pressure
4. Vary Time
5. Evaluate
D. Polish
1. Select Wheel
2. Select Grinding Media
3. Select Grit Size
4. Vary Pressure
5. Vary Time
6. Evaluate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Aug
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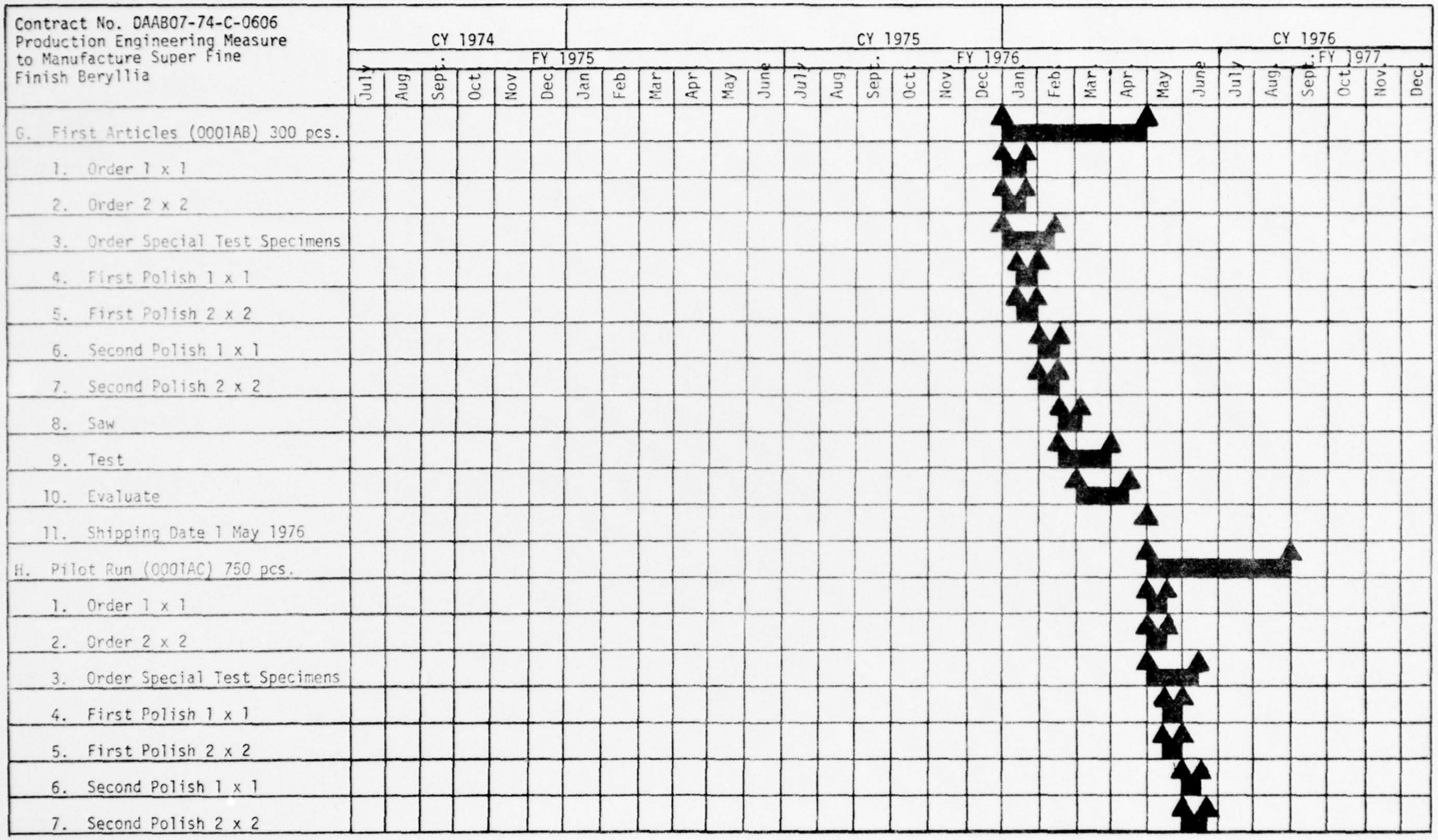
E. Definition of Process
1. Test Run on 1 x 1
2. Test Run on 2 x 2
3. Saw
4. Evaluate
F. Engineering Samples (0001AA) 75 pcs.
1. Order 1 x 1
2. Order 2 x 2
3. Order Special Test Specimens
4. First Polish 1 x 1
5. First Polish 2 x 2
6. Second Polish 1 x 1
7. Second Polish 2 x 2
8. Saw
9. Test
10. Evaluate
11. Report (B001AA)
12. Shipping Date 25 June 1975
13. Final Specification


Contract No. DAAB07-74-C-0606
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Finish Beryllia
CY 1974
CY 1975
CY 1976
FY 1975
FY 1976
FY 1977
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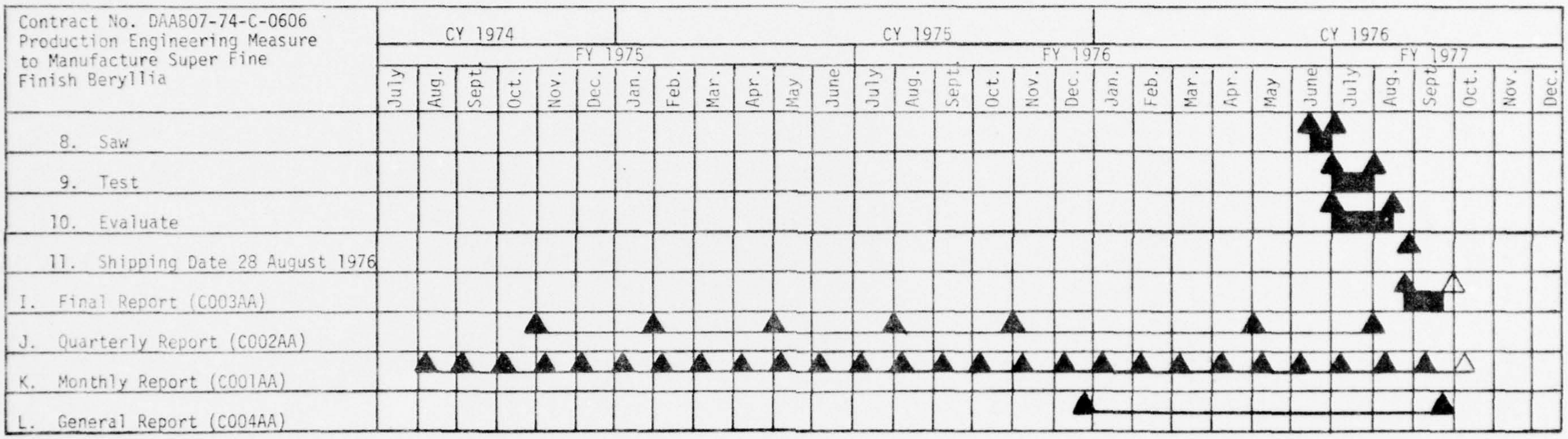
June
G. First Articles (0001AB) 300 pcs.
1. Order 1 x 1
2. Order 2 x 2
3. Order Special Test Specimens
4. First Polish 1 x 1
5. First Polish 2 x 2
6. Second Polish 1 x 1
7. Second Polish 2 x 2
8. Saw
9. Test
10. Evaluate
11. Shipping Date 1 May 1976
H. Pilot Run (0001AC) 750 pcs.
1. Order 1 x 1
2. Order 2 x 2
3. Order Special Test Specimens
4. First Polish 1 x 1
5. First Polish 2 x 2
6. Second Polish 1 x 1
7. Second Polish 2 x 2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CY 1974
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July Aug. Sept. Oct. Nov. Dec. Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec. Jan. Feb. Mar. Apr. May June July Aug. Sept. Oct. Nov. Dec.
8. Saw
9. Test
10. Evaluate
11. Shipping Date 28 August 1976
I. Final Report (C003AA)
J. Quarterly Report (C002AA)
K. Monthly Report (C001AA)
L. General Report (C004AA)

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